Fixing vs. Rotating Hydrothermal Solution Synthesis of Olivine $LiFePO_4$-C Cathode Materials for Li-ion Rechargeable Batteries

Kumaran Vediappan[1, 2], Abdelbast Guerfi[1], George P. Demopoulos[2], Julie Trottier[1], Catherine Gagnon[1], Pierre Hovington[1] and Karim Zaghib[1]*

[1]Institut de Recherche d'Hydro-Québec (IREQ), 1800 Bd Lionel-Boulet, Varennes (QC), Canada J3X 1S1
[2]Department of Mining and Materials Engineering, McGill University, Wong Building, 3610 University Street, Montreal, QC H3A 2B2, Canada
*Corresponding Author: zaghib.karim@ireq.ca

Li-ion batteries constitute promising electrochemical power systems, because they have higher operating voltage and higher power/energy density than other battery systems. Until now, various methods have been developed for the preparation of $LiFePO_4$ cathode materials, such as sol-gel method, co-precipitation, mechano-chemical method, spray technology, and solid-state reactions. However, all the above methods have some difficulties and limits when it comes to commercial production. Hence the hydrothermal method may offer a simpler solution in solving the practical and scale-up problems [1]. Over the last 10 years, many research groups have focused on hydrothermal synthesis (HTS) of electrode materials for lithium-ion batteries [2].

In this research work, $LiFePO_4$-C cathode materials were prepared by hydrothermal synthesis by employing two different mixing methods: the fixing method in which no solution agitation is applied and the rotating method in which solution agitation is applied. In both cases the same types and ratios of precursors were used, namely: $LiOH \cdot H_2O$, $FeSO_4 \cdot 7H_2O$, H_3PO_4 (85wt %), and ascorbic acid as carbon source in stoichiometric molar ratios of Li, Fe, P, and C (3:1:1:0.2). Annealing was done at 700°C under nitrogen atmosphere as previously described in [2, 3]. The two different synthesized cathode powders are discussed and compared in terms of structural, surface morphological and electrochemical characterizations via X-ray diffraction spectroscopy (XRD), scanning electron microscopy (SEM) (as shown in Fig. 1) and electrochemical studies.

The electrochemical performance was analyzed in the cut-off voltage range of 2.0 to 4.0 V (vs. Li/Li^+) `at room temperature as well as 55°C. The $LiFePO_4$-C electrodes prepared by the rotating method were found to exhibit higher discharge capacities of 137.6mAh/g at C/12 when compared to the fixing method prepared electrodes (106.2mAh/g). Also, other higher current rates of rotating method prepared electrodes were close to the same specific capacity as that at C/12. Furthermore, the rotating method synthesized cathode materials retained more than 98% of the coulombic efficiency even after 20 cycles, this attributed to less aggregated smaller particles with high surface area and less impurities. Hence, the rotating solution synthesis method is very effective in producing smaller cathode particles at high yield with very good capacity retention characteristics for use in rechargeable Li-ion batteries.

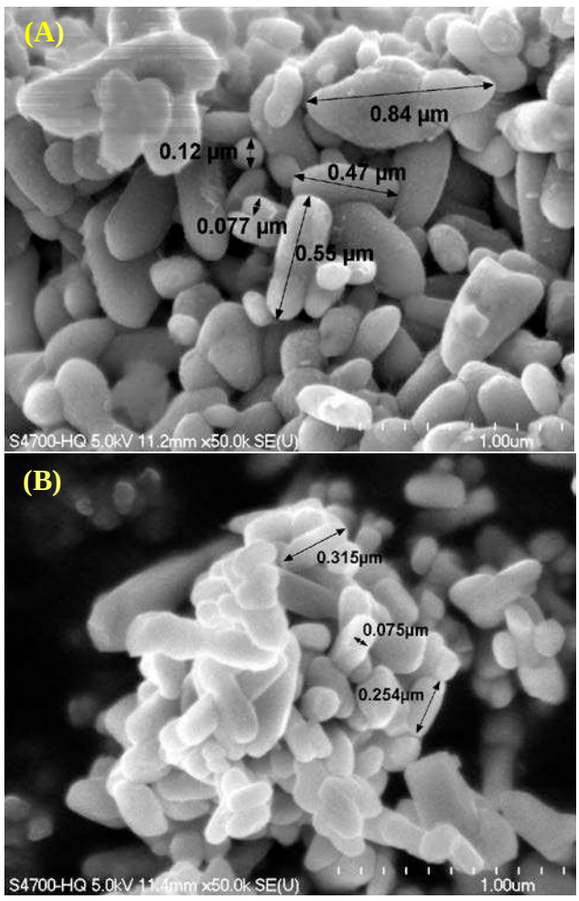

Figure 1. SEM images of hydrothermally synthesized $LiFePO_4$-C powders through (A) Fixing solution method, (B) Rotating solution method.

References

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